State of California
Air Resources Board

Method 13A

Determination of Total Fluoride Emissions from Stationary Sources (SPADNS Zirconium Lake Method)

Adopted: March 28, 1986
Amended: _____

Note: this document consists of the text of the proposed amendment to Method 13A. Proposed deletions are noted by graphic screen and proposed additions are noted by underline.

Method 13A - Determination of Total Fluoride Emissions from Stationary Sources (SPADNS Zirconium Lake Method)

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability. This method applies to the determination of fluoride (F) emissions from sources as specified in the regulations. It does not measure fluorocarbons, such as fFreons.

1.2 Principle. Gaseous and particulate F are withdrawn isokinetically from the source and collected in water and on a filter. The total F is then determined by the SPADNS Zirconium Lake Colorimetric method.

Any modification of this method beyond those expressly permitted shall be considered a major modification subject to the approval of the Executive Officer. The term Executive Officer as used in this document shall mean the Executive Officer of the Air Resources Board (ARB), or his or her authorized representative.

2. RANGE AND SENSITIVITY

The range of this method is 0 to 1.4 µg F/ml. Sensitivity has not been determined.

3. INTERFERENCES

Large quantities of chloride will interfere with the analysis, but this interference can be prevented by adding silver sulfate into the distillation flask (see Section 7.3.4). If chloride ion is present, it may be easier to use the Specific Ion Electrode Method (Method 13B). Grease on sample-exposed surfaces may cause low F results due to adsorption.

4. PRECISION, ACCURACY, AND STABILITY

4.1 Precision. The following estimates are based on a collaborative test done at a primary aluminum smelter. In the test, six laboratories each sampled the stack simultaneously using two sampling trains for a total of 12 samples per sampling run. Fluoride concentrations encountered during the test ranged from 0.1 to 1.4 mg F/m3m^3. The within-laboratory and between-laboratory standard deviations, which include sampling and analysis errors, were 0.044 mg F/m^3 with 60 degrees of freedom and 0.064 mg F/m^3 with five degrees of freedom, respectively.

4.2 Accuracy. The collaborative test did not find any bias in the analytical method.

4.3 Stability. After the sample and colorimetric reagent are mixed, the color formed is stable for approximately 2 hours. A 3^o C temperature difference between the sample and standard solutions produces an error of approximately 0.005 mg F/liter. To avoid this error, the absorbencies of the sample and standard solutions must be measured at the same temperature.

5. APPARATUS

5.1 Sampling Train. A schematic of the sampling train is shown in Figure 13A-1; it is similar to the Method 5 train except the filter position is interchangeable. The sampling train consists of the following components:

5.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1, 2.1.3, 2.1.4, 2.1.6, 2.1.8, 2.1.9, and 2.1.10, respectively. When moisture condensation is a problem, the filter heating system is used.

5.1.2 Probe Liner. Borosilicate glass or 316 stainless steel. When the filter is located immediately after the probe, the tester may use a probe heating system to prevent filter plugging resulting from moisture condensation, but the tester shall not allow the temperature in the probe to exceed 120 ± 14 ^{o}C (248 ± 25 ^{o} F).

5.1.3 Filter Holder. With positive seal against leakage from the outside or around the filter. If the filter is located between the probe and first impinger, use borosilicate glass or stainless steel with a 20-mesh stainless steel screen filter support and a silicone rubber gasket; do not use a glass frit or a sintered metal filter support. If the filter is located between the third and fourth impingers, the tester may use borosilicate glass with a glass frit filter support and a silicone rubber gasket. The tester may also use other materials of construction with approval from the Control Agency's authorized representativeExecutive Officer.

5.1.4 Impingers. Four impingers connected as shown in Figure 13A-1 with ground-glass (or equivalent), vacuum-tight fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm (1/2-in.) ID glass tube extending to 1.3 cm (1/2 in.) from the bottom of the flask. For the second impinger, use a Greenburg-Smith impinger with the standard tip. The tester may use modifications (e.g., flexible connections between the impingers or materials other than glass), subject to the approval of the Control Agency's authorized representativeExecutive Officer. Place a thermometer, capable of measuring temperature to within 1 C (2 F)1^{o}C (2^{o}F), at the outlet of the fourth impinger for monitoring purposes.

5.2 Sample Recovery. The following items are need:

5.2.1 Probe-liner and Probe-Nozzle Brushes, Wash Bottles, Graduated Cylinder and/or Balance, Plastic Storage Containers, Rubber Policeman, and Funnel. Same as Method 5, Sections 2.2.1, 2.2.2 and 2.2.5 to 2.2.8, respectively.

5.2.2 Sample Storage Container. Wide-mouth, high-density polyethylene bottles for impinger water samples, 1 liter.

5.3 Analysis. The following equipment is needed:

5.3.1 Distillation Apparatus. Glass distillation apparatus assembled as shown in Figure 13A-2.

5.3.2 Bunsen Burner.

5.3.3 Electric Muffle Furnace. Capable of heating to 600 ^{o}C.

5.3.4 Crucibles. Nickel, 75- to 100-ml.

5.3.5 Beakers. 500-ml 200-ml and 1500-ml.

5.3.6 Volumetric Flasks. 50-ml, 100 ml, 250 ml, 500 ml, and 1 liter.

5.3.7 Erlenmeyer Flasks or Plastic Bottles. 500-ml.

5.3.8 Constant Temperature Bath. Capable of maintaining a constant temperature of ±1.0^{o}C at room temperature conditions.

5.3.9 Balance. 300-g capacity, to measure to ±0.5 g.

5.3.10 Spectrophotometer. Instrument that measures absorbance at 570 nm and provides at least a 1-cm light path.

5.3.11 Spectrophotometer Cells. 1-cm pathlength.

6. REAGENTS

Use ACS reagent-grade chemicals, or equivalent, unless otherwise specified.

NOTE: Mention of company or product names does not constitute endorsement by the Air Resources Board.

6.1 Sampling. Use ACS reagent-grade chemicals or equivalent, unless otherwise specified. The reagents used in sampling are as follows:

6.1.1 Filters.

6.1.1.1 If the filter is located between the third and fourth impingers, use a Whatman No. 1 filter, or equivalent, sized to fit the filter holder.

6.1.1.2 If the filter is located between the probe and first impinger, use any suitable medium (e.g., paper, organic membrane) that conforms to the following specifications: (1) The filter can withstand prolonged exposure to temperatures up to 135^{o}C (275^{o}F). (2) The filter has at least 95 percent collection efficiency (<5 percent penetration) for 0.3 umμm dioctyl phthalate smoke particles. Conduct the filter efficiency test before the test series, using ASTM Standard Method D 2986-71, or use test data from the supplier's quality control program. (3) The filter has a low F blank value (<0.015 mg F/cm^{2} of filter area). Before the test series, determine the average F blank value of at least three filters (from the lot to be used for sampling) using the applicable procedures described in Sections 7.3 and 7.4 of this method. In general, glass fiber filters have high and/or variable F blank values, and will not be acceptable for use.

6.1.2 Water. Deionized distilled, to conform to ASTM Specification D 1193-74, Type 3. If high concentrations of organic matter are not expected to be present, the analyst

may delete the potassium permanganate test for oxidizable organic matter.

6.1.3 Silica Gel, Crushed Ice, and Stopcock Grease. Same as Method 5, Section 3.1.2, 3.1.4, and 3.1.5, respectively.

6.2 Sample Recovery. Water, from same container as described in Section 6.1.2, is needed for sample recovery.

6.3 Sample Preparation and Analysis. The reagents needed for sample preparation and analysis are as follows:

6.3.1 Calcium Oxide (Ca0). Certified grade containing 0.005 percent F or less.

6.3.2 Phenolphthalein Indicator. Dissolve 0.1 g of phenolphthalein in a mixture of 50 ml of 90 percent ethanol and 50 ml of deionized distilled water.

6.3.3 Silver Sulfate (Ag_2SO_4).

6.3.4 Sodium Hydroxide (Na0H), Pellets.

6.3.5 Sulfuric Acid (H_2SO_4), Concentrated.

6.3.6 Sulfuric Acid, 25 Percent (v/v). Mix 1 part of concentrated H_2SO_4 with 3 parts of deionized distilled water.

6.3.7 Filters. Whatman No. 541, or equivalent.

6.3.8 Hydrochloric Acid (HCl), Concentrated.

6.3.9 Water. From same container as described in section 6.1.2. Same as in Section 6.1.2.

6.3.10 Fluoride Standard Solution, 0.01 mg F/ml. Dry in an oven at 110°C for at least 2 hours. Dissolve 0.2210 g of NaF in 1 liter of deionized distilled water. Dilute 100 ml of this solution to 1 liter with deionized distilled water.

6.3.11 SPADNS Solution [4,5 dihydroxyy-3-(p-sulfophenylazo)-2,7- naphthalene-disulfonic acid trisodium salt]. Dissolve 0.960 ± 0.010 g of SPADNS reagent in 500 ml deionized distilled water. If stored in a well-sealed bottle protected from the sunlight, this solution is stable for at least 1 month.

6.3.12 Spectrophotometer Zero Reference Solution. Prepare daily. Add 10 ml of SPADNS (6.3.1) solution to 100 ml deionized distilled water, and acidify with a solution prepared by diluting 7 ml of concentrated HCl to 10 ml with deionized, distilled water.

6.3.13 SPADNS Mixed Reagent. Dissolve 0.135 ± 0.005 g of zirconyl chloride octahydrate ($Zr0Cl_2.8H_2O$) in 25 ml of deionized distilled water. Add 350 ml of concentrated HCl, and dilute to 500 ml with deionized, distilled water. Mix equal volumes of this solution and SPADNS solution to form a single reagent. This reagent

is stable for at least 2 months.

7. PROCEDURE

7.1 Sampling. Because of the complexity of this method, testers should be trained and experienced with the test procedures to assure reliable results.

7.1.1 Pretest Preparation. Follow the general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

7.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, Section 4.1.2, except the nozzle size selected must maintain isokinetic sampling rates below 28 liters/min (1.0 cfm).

7.1.3 Preparation of Collection Train. Follow the general procedure given in Method 5, Section 4.1.3, except for the following variations:

Place 100 ml of deionized distilled water in each of the first two impingers, and leave the third impinger empty. Transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger.

Assemble the train as shown in Figure 13A-1 with the filter between the third and fourth impingers. Alternatively, if a 20-mesh stainless steel screen is used for the filter support, the tester may place the filter between the probe and first impinger. The tester may also use a filter heating system to prevent moisture condensation, but shall not allow the temperature to exceed 120 ± 14°C (248 ± 25°F). Record the filter location on the data sheet.

7.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5, Sections 4.1.4.1(Pretest Leak Check), 4.1.4.2(Leak Check During the Sample Run), and 4.1.4.3(Post-Test Leak Check).

7.1.5 Fluoride Train Operation. Follow the general procedure given in Method 5, Section 4.1.5, keeping the filter and probe temperatures (if applicable) at 120 ± 14°C (248 ± 25°F) and isokinetic sampling rates below 28 liters/min (1.0 cfm). For each run, record the data required on a data sheet such as the one shown in Method 5, Figure 5-2.

7.2 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle, and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, because a vacuum would form in the filter holder, thus drawing impinger water backwards.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Remove the filter assembly, wipe off the silicone grease from the filter holder inlet, and cap this inlet. Remove the umbilical cord from the last

impinger, and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and any open impinger inlets and outlets. The tester may use ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and filter-impinger assembly to an area that is clean and protected from the wind so that the chances of contaminating or losing the sample is minimized.

Inspect the train before and during disassembly, and note any abnormal conditions. Treat the samples as follows:

7.2.1 Container No. 1 (Probe, Filter, and Impinger Catches). Using a graduated cylinder, measure to the nearest ml, and record the volume of the water in the first three impingers; include any condensate in the probe in this determination. Transfer the impinger water from the graduated cylinder into this polyethylene container. Add the filter to this container. (The filter may be handled separately using procedures subject to the Control Agency's authorized representativeExecutive Officer's approval.) Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, clean all sample-expose surfaces (including the probe nozzle, probe fitting, probe liner, first three impingers, impinger connectors, and filter holder) with deionized distilled water. Use less than 500 ml for the entire wash. Add the washings to the sample container. Perform the water rinses as follows:

Carefully remove the probe nozzle and rinse the inside surface with deionized distilled water from a wash bottle. Brush with a Nylon bristle brush, and rinse until the rinse shows no visible particles, after which make a final rinse of the inside surface. Brush and rinse the inside parts of the Swagelok fitting with deionized distilled water in a similar way.

Rinse the probe liner with deionized distilled water. While squirting the water into the upper end of the probe, tilt and rotate the probe so that all inside surfaces will be wetted with water. Let the water drain from the lower end into the sample container. The tester may use a funnel (glass or polyethylene) to aid in transferring the liquid washes to the container. Follow the rinse with a probe brush. Hold the probe in an inclined position, and squirt deionized distilled water into the upper end as the probe brush is being pushed with a twisting action through the probe. Hold the sample container underneath the lower end of the probe, and catch any water and particulate matter that is brushed from the probe. Run the brush through the probe three times or more. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with deionized distilled water, and quantitatively collect these washings in the sample container. After the brushing, make a final rinse of the probe as described above.

It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

Rinse the inside surface of each of the first three impingers (and connecting glassware) three separate times. Use a small portion of deionized distilled water for each rinse, and brush each sample-exposed surface with a Nylon bristle brush, to ensure recovery of fine particulate matter. Make a final rinse of each surface and of the brush.

After ensuring that all joints have been wiped clean of the silicone grease, brush and rinse with deionized distilled water the inside of the filter holder (front-half only, if filter is positioned between the third and fourth impingers). Brush and rinse each surface three times or more if needed. Make a final rinse of the brush and filter holder.

After all water washings and particulate matter have been collected in the sample container, tighten the lid so that water will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container clearly to identify its contents.

7.2.2 Container No. 2 (Sample Blank). Prepare a blank by placing an unused filter in a polyethylene container and adding a volume of water equal to the total volume in Container No. 1. Process the blank in the same manner as for Container No. 1.

7.2.3 Container No. 3 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container, and seal. The tester may use a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, the tester may follow the analytical procedure for Container No. 3 in Section 7.4.2.

7.3 Sample Preparation and Distillation. (Note the liquid levels in Containers No. 1 and No. 2, and confirm on the analysis sheet whether leakage occurred during transport. If noticeable leakage had occurred, either void the sample or use methods, subject to the approval of the Control Agency's authorized representativeExecutive Officer, to correct the final results.) Treat the contents of each sample container as described below:

7.3.1 Container No. 1 (Probe, Filter, and Impinger Catches). Filter this container's contents, including the sampling filter, through Whatman No. 541 filter paper, or equivalent, into a 1500-ml beaker.

7.3.1.1 If the filtrate volume exceeds 900 ml, make the filtrate basic (red to phenolphthalein) with NaOH, and evaporate to less than 900 ml.

7.3.1.2 Place the filtered material (including sampling filter) in a nickel crucible, add a few ml of deionized distilled water, and macerate the filters with a glass rod.

Add 100 mg CaO to the crucible, and mix the contents thoroughly to form a slurry. Add two drops of phenolphthalein indicator. Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely. During the evaporation of the water, keep the slurry basic (red to phenolphthalein) to avoid loss of F. If the indicator turns colorless (acidic) during the evaporation, add Ca0 until the color turns red again.

After evaporation of the water, place the crucible on a hot plate under a hood,

and slowly increase the temperature until the Whatman No. 541 and sampling filters char. It may take several hours to char the filters completely.

Place the crucible in a cold muffle furnace. Gradually (to prevent smoking) increase the temperature to 600° C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace, and allow to cool.

Add approximately 4 g of crushed NaOH to the crucible, and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600° C.

Remove the sample from the furnace, and cool to ambient temperature. Using several rinsings of warm deionized distilled water, transfer the contents of the crucible to the beaker containing the filtrate. To assure complete sample removal, rinse finally with two 20-ml portions of 25 percent H_2SO_4, and carefully add to the beaker. Mix well, and transfer to a 1-liter volumetric flask. Dilute to volume with deionized distilled water, and mix thoroughly. Allow any undissolved solids to settle.

7.3.2 Container No. 2 (Sample Blank). Treat in the same manner as described in Section 7.3.1 above.

7.3.3 Adjustment of Acid/Water Ratio in Distillation Flask. (Use a protective shield when carrying out this procedure.) Place 400 ml of deionized distilled water in the distillation flask, and add 200 ml of concentrated H_2SO_4. (Caution: Observe standard precautions when mixing H_2SO_4 with water. Slowly add the acid to the flask with constant swirling.) Add some soft glass beads and several small pieces of broken glass tubing, and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175°C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.3.4 Distillation. Cool the contents of the distillation flask to below 80°C. Pipet an aliquot of sample containing less than 10.0 mg F directly into the distillation flask, and add deionized distilled water to make a total volume of 220 ml added to the distillation flask. (To estimate the appropriate aliquot size, select an aliquot of the solution, and treat as described in Section 7.4.1. This will be an approximation of the F content because of possible interfering ions.)

NOTE: If the sample contains chloride, add 5 mg of Ag_2SO_4 to the flask for every mg of chloride.

Place a 250-ml volumetric flask at the condenser exit. Heat the flask as rapidly as possible with a Bunsen burner, and collect all the distillate up to 175°C. During heatup, play the burner flame up and down the side of the flask to prevent bumping. Conduct the distillation as rapidly as possible (15 minutes or less). Slow distillations have been found to produce low F recoveries. Caution: Be careful not to exceed 175°C to avoid causing H_2SO_4 to distill over.

If F distillation in the mg range is to be followed by a distillation in the fractional mg range, add 220 ml of deionized distilled water, and distill it over as in the acid adjustment step to remove residual F from the distillation system.

The tester may use the acid in the distillation flask until there is carry-over of interferences or poor F recovery. Check for these every tenth distillation using a deionized distilled water blank and a standard solution. Change the acid whenever the F recovery is less than 90 percent or the blank value exceeds 0.1 ugµg/ml.

7.4 Analysis.

7.4.1 Containers No. 1 and No. 2. After distilling suitable aliquots from Containers No. 1 and No. 2 according to Section 7.3.4, dilute the distillate in the volumetric flasks to exactly 250 ml with deionized distilled water, and mix thoroughly. Pipet a suitable aliquot of each sample distillate (containing 10 to 40 ugµg F/ml) into a beaker, and dilute to 50 ml with deionized distilled water. Use the same aliquot size for the blank. Add 10 ml of SPADNS mixed reagent (Section 6.3.13), and mix thoroughly.

After mixing, place the sample in a constant-temperature bath containing the standard solutions (see Section 8.2) for 30 minutes before reading the absorbance on the spectrophotometer.

Set the spectrophotometer to zero absorbance at 570 nm with the reference solution (Section 6.3.12), and check the spectrophotometer calibration with the standard solution. Determine the absorbance of the samples, and determine the concentration from the calibration curve. If the concentration does not fall within the range of the calibration curve, repeat the procedure using a different size aliquot.

7.4.2 Container No. 3 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. The tester may conduct this step in the field.

8. CALIBRATION

Maintain a laboratory log of all calibrations.

8.1 Sampling Train. Calibrate the sampling train components according to the indicated sections in Method 5: Probe Nozzle (Section 5.1); Pitot Tube (Section 5.2); Metering Syetem (Section 5.3); Probe Heater (Section 5.4); Temperature Gauges (Secton 5.5); Leak Check of Metering System (Section 5.6); and Barometer (Section 5.7). Calibrate the probe nozzle, pitot tube, metering system, probe heater, temperature gauges, and barometer according to Method 5, Sections 5.1, 5.2, 5.3, 5.4, 5.5, and 5.7, respectively. Conduct the leak-check of the metering system according to Method 5, Section 5.6.

8.2 Spectrophotometer. Prepare the blank standard by adding 10 ml of SPADNS mixed reagent to 50 ml of deionized distilled water. Accurately prepare a series of standards from the 0.01 mg F/ml standard fluoride solution (Section 6.3.10) by diluting 0, 2, 4, 6, 8, 10, 12, and 14 ml to 100 ml with deionized, distilled water. Pipet 50 ml from each solution, and transfer each to a separate 200-ml beaker. Then add 10 ml of SPADNS mixed reagent to each. These standards will contain 0, 10, 20, 30, 40, 50, 60, and 70 ugµg F (0 to 1.4 ugµg/ml), respectively.

After mixing, place the reference standards and reference solution in a constant temperature bath for 30 minutes before reading the absorbance with the

spectrophotometer. Adjust all samples to this same temperature before analyzing.

With the spectrophotometer at 570 nm, use the reference solution (Section 6.3.12) to set the absorbance to zero.

Determine the absorbance of the standards. Prepare a calibration curve by plotting ugμg F/50 ml versus absorbance on linear graph paper. Prepare the standard curve initially and thereafter whenever the SPADNS mixed reagent is newly made. Also, run a calibration standard with each set of samples and, if it differs from the calibration curve by ±2 percent, prepare a new standard curve.

9. CALCULATIONS

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation. Other forms of the equations may be used, provided that they yield equivalent results.

9.1 Nomenclature.

A_d = Aliquot of distillate taken for color development, ml.

A_t = Aliquot of total sample added to still, ml.

B_{ws} = Water vapor in the gas stream, portion by volume.

C_s = Concentration of F in stack gas, mg/ft^3, dry basis, corrected to standard conditions of 760 mm Hg (29.92 in. Hg) and 293^o K (29.92 in. Hg and 528^o R), mg/m^3 (mg/ft^3).

F_t = Total F in sample, mg.

ugμg F = Concentration from the calibration curve, ugμg.

T_m = Absolute average dry gas meter temperature (see Figure 5-2 of Method 5), ^{o}K (^{o}R).

T_s = Absolute average stack gas temperature (see Figure 5-2 of Method 5), ^{o}K (^{o}R).

V_d = Volume of distillate as diluted, ml.

$V_{m(std)}$ = Volume of gas sample as measured by DGM at standard conditions, dscm (dscf).

V_t = Total volume of F sample, after final dilution, ml.

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to at standard conditions, scm (scf)

9.2 Average DGM Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5-2 of Method 5).

9.3 Dry Gas Volume. Calculate Vm(std), and adjust for leakage, if necessary, using the equation of in sSection 6.3 of Method 5.

9.4 Volume of Water Vapor and Moisture Content. Calculate the volume of water vapor $V_{w(std)}$ and moisture content B_{ws} from the data obtained in this method (Figure 13A-l); use Equations 5-2 and 5-3 of Method 5.

9.5 Concentration.

9.5.1 Total Fluoride in Sample. Calculate the amount of F in the sample using the following equation:

equation to be deleted:

$$F_t = \frac{10^{-3}\ V_t\ V_d}{A_t\ A_d}(ug\ F) \qquad \text{Eq. 13A-1}$$

equation to be added:

$$F_t = \frac{10^{-3}\ V_t\ V_d}{A_t\ A_d}(\mu g\ F) \qquad \text{Eq. 13A-1}$$

9.5.2 Fluoride Concentration in Stack Gas. Determine the F concentration in the stack gas using the following equation:

$$C_s = \frac{K\ F_t}{V_{m(std)}} \qquad \text{Eq. 13A-2}$$

Where:

K = 1.00 m^3/m^3 if $V_{m(std)}$ is expressed in metric units.

= 35.31 ft^3/m^3 if $V_{m(std)}$ is expressed in English units.

9.6 Isokinetic Variation and Acceptable Results. Use Method 5, Sections 6.11.1 and 6.11.2.

10. BIBLIOGRAPHY

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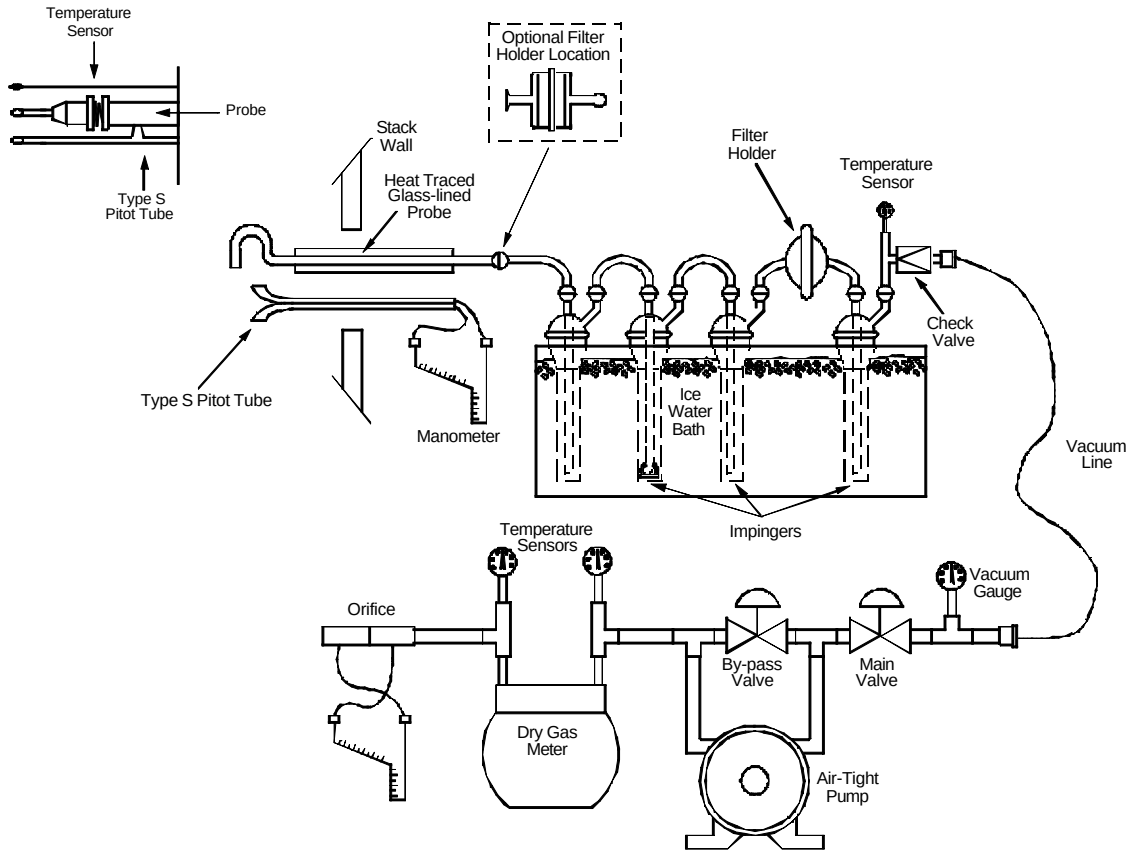

Figure 13A-1. Fluoride Sampling Train

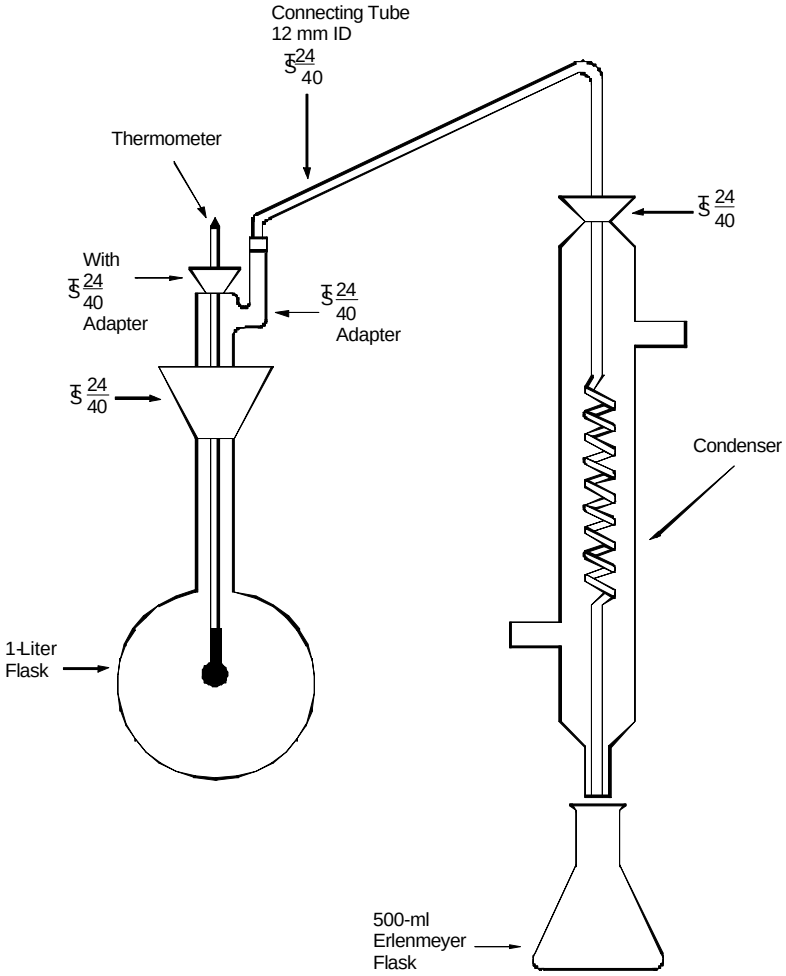

Figure 13A-2. Fluoride Distillation Apparatus.